PATENT SPECIFICATION 1,153,483

NO DRAWINGS.

Inventor:—JOSEPH SIDNEY WEST.

1,153,483

Date of filing Complete Specification: 21 Feb., 1967.

Application Date: 31 March, 1966. No. 14212/66.

Complete Specification Published: 29 May, 1969.

© Crown Copyright 1969.

Index at Acceptance:—**C1** AE4R1.

Int. Cl.:—**C 01 b 21/16.**

COMPLETE SPECIFICATION.

Hydrazine.

We, FISONS INDUSTRIAL CHEMICALS LIMITED formerly known as WHIFFEN & SONS LIMITED, a British Company, of Willows Works, Derby Road, Loughborough, Leicestershire, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to the preparation of hydrazine from urea.

It is well known that hydrazine can be obtained by oxidising urea under appropriate conditions with sodium hypochlorite in the presence of a large excess of sodium hydroxide. However the yields are low and the presence of a large excess of sodium hydroxide leads to relatively low concentrations of hydrazine and makes distillation difficult due to excessive foaming. It has now been found that comparatively high yields can be obtained using substantially the theoretical amount of alkali if the process is operated in two stages with close control of the proportions of reactants.

Accordingly the present invention provides a process for the preparation of hydrazine which comprises reacting in an aqueous medium at least 1·0 mole, preferably 1·0 to 1·1, moles of urea with 1·0 mole of sodium hypochlorite in the presence of m moles of alkali metal hydroxide, preferably sodium hydroxide, where m is a figure in the range 0·1 to 0·3, whereby a solution containing monochlorurea is formed and subsequently reacting one mole of the monochlorurea with x moles of alkali metal hydroxide wherein m + x is in the range 1·9 to 2·1 whereby a solution containing hydrazine is obtained.

Preferably the process is operated in a relatively concentrated solution. In this connection the urea is preferably fed to the reaction as a solution in water containing 30% to 50% by weight of urea; the alkali metal hydroxide, preferably sodium hydroxide, is preferably fed to the reaction as a solution in water containing 40% to 50% by weight of alkali metal hydroxide, for example 50% by weight of alkali metal hydroxide; and the sodium hypochlorite is preferably fed to the reaction as a solution in water containing 10% to 15% by weight of sodium hypochlorite.

The first stage of the process of the present invention, that is the treatment of urea with hypochlorite in the presence of sodium hydroxide, is desirably carried out at as low a temperature as is practicable and is preferably carried out at a temperature in the range – 10° C. to 10° C. The second stage of the process of the present invention, that is the treatment of monochlorurea with sodium hydroxide, is desirably carried out at an elevated temperature and is preferably carried out at a temperature in the range 90° C. to 110° C. In the second stage it is desirable to raise the temperature as rapidly as possible. In this stage a short residence time is advantageous for example about 30—60 seconds at 100° C.

The pH at which the first stage is operated is preferably maintained within the range 8 to 11.

In order to improve yields it is preferred to carry out the second stage of the process in the presence of gelatin or glue preferably at a concentration in the range 0·2 to 1·0% by weight in the reaction mixture.

The following example, in which parts are by weight, is given to illustrate the process of the present invention.